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(54) Regenerated cellulose fibres

(57) Highly absorbent regenerated cellulose fibres having water imbibitions which are usually in the range 150 to 300 *per cent* may be made by spinning cellulose fibres with a lumen and incorporating a dispersed polymer and/or substituent groups on the cellulose to increase the absorbency. The lumen may be produced by incorporating a blowing agent such as sodium carbonate in the viscose and may be either in expanded form to give a tubular structure or at least partially collapsed to give a multi-limbed structure. The polymer additive may for example be sodium polyacrylate, poly(vinyl pyrrolidone) or sodium carboxymethyl cellulose, and is suitably used in a concentration less than 5 *per cent* by weight. The fibres are useful in making swabs, dressings and catamenial tampons.

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SPECIFICATION

Regenerated cellulose fibres

This invention relates to regenerated cellulose fibres which are highly absorbent to liquids, and which are of particular benefit in end uses where this property is necessary including towelling, diapers and medical and sanitary products. Such products are currently being made from cotton and conventional regenerated cellulose fibres, and recently from so-called 'alloy' fibres.

Alloy fibres are basically regenerated cellulose fibres made by mixing a conventional viscose with a more absorbent polymer such as poly(vinyl pyrrolidone) or cyanoethylated viscose. By this technique the water imbibition of the fibres can be increased from the 90 *per cent* level of conventional regenerated cellulose fibres up to in excess of 200 *per cent*, depending on the concentration of the more absorbent polymer. As usual, the benefit of increased absorbency is paid for in terms of additional cost and a reduction in other fibre properties.

According to this invention, regenerated cellulose fibres comprise a cellulose wall defining a lumen, the cellulose wall incorporating substituent groups on the cellulose and/or a dispersed polymeric material, which substituent groups or polymeric material increase the liquid absorbency of the fibres.

The term 'fibres' includes both continuous filaments and staple fibres.

The lumen in the fibres of the invention may be in expanded or collapsed or partially collapsed form. The presence of the lumen in the fibres gives them a greatly increased liquid absorbency as compared with solid fibres of the same denier. For example, an expanded lumen which makes the fibre structure substantially tubular can impart a water imbibition of about 125 *per cent* compared with the 90 *per cent* of a solid fibres. Fibres having collapsed or partially collapsed lumens may have water imbibitions as high as 200 *per cent* or more although a substantial portion of this increase is due to interstitial water held by the multi-limbed cross-section which is formed when the lumen collapses. With this raised 'base-line' it is possible to increase the water imbibition of the fibres to the desired level using either fewer substituent groups or less of the dispersed polymeric material than would be the case with a solid fibre.

The fibres of the invention preferably have a water imbibition of at least 150 *per cent*. For most end uses the desired water imbibition will be in the range 150 to 300 *per cent*.

Suitable substituent groups for the cellulose include carboxyethyl groups which are the predominant groups formed by hydrolysis of cyanoethyl groups during regeneration. The cyanoethyl groups may be formed by adding acrylonitrile to viscose or by adding carbon disulphide and acrylonitrile to alkali cellulose to effect simultaneous xanthation and cyanoethylation. Hydroxyethyl substituent groups are also suitable and may be similarly formed, using ethylene oxide instead of acrylonitrile.

The degree of substitution is controllable and so can be selected to confer the desired level of absorbency. Of course it is not necessary to use the substituted viscose by itself and it can be mixed with unsubstituted viscose to adjust the absorbency to the level required. This allows standardisation of the substitution process at the optimum conditions whilst retaining the freedom to vary the fibre absorbency.

Suitable polymeric materials for dispersion in the viscose, preferably from aqueous solution, include: cellulose ethers particularly carboxyalkyl celluloses such as sodium carboxymethyl cellulose; alkali metal or salts of polyacrylic acid particularly sodium polyacrylate, and alkali metal or ammonium salts of a copolymer of acrylic acid and methacrylic acid; an N-vinyl amide polymer, particularly poly(vinyl pyrrolidone); carboxyalkylated starch; and poly(ethylene glycol). The polymer solutions may be mixed with the viscose in a blender or simply injected into the viscose feed lines to the spinning jets.

The water imbibition of the fibres increases as the amount of polymeric material incorporated in the cellulose is increased. However, we have found that it is usually possible to boost the water imbibition to the desired level in the range of 150 to 300 *per cent* using additive amounts of polymeric material of less than 5 *per cent* by weight based on the weight of the cellulose. It is preferred to use such small amounts because the cost of the additive is reduced and the effect on the mechanical properties of the fibres is minimised.

Regenerated cellulose fibres having a lumen may be produced by incorporating a blowing agent, for example sodium carbonate, into the viscose so that the spun viscose filaments inflate in the regenerating bath. The viscose formulation, the composition of the regenerating bath and the spinning conditions can be chosen to give various rates of regeneration and inflation. In this way, the lumen may be maintained in an expanded form to give a substantially tubular fibre as described in our British Patent No. 1,283,529, or it may be allowed to inflate more rapidly with subsequent collapse or partial collapse, to give a multi-limbed cross-section as described in our British Patents Nos. 1,333,047 and 1,393,778.

The water imbibition of the fibres is measured by taking a 1 gm sample of fibres, soaking it in water at a temperature of 20°C. for 30 minutes, centrifuging it at a force of 1,000 gravities for

5 minutes, weighting it (W_1), drying it at a temperature of 110°C. for 2.5 hours, and finally reweighing it (W_2). The water imbibition, expressed as a percentage, is then equal to

$$\frac{W_1-W_2}{W_2} \times 100$$

The invention includes a fabric or pad, particularly a swab or dressing, comprising the regenerated cellulose fibres of the invention. It further includes a catamenial tampon comprising the regenerated cellulose filaments of the invention.

The invention is illustrated by the following Examples in which percentages are by weight:—

Example 1

A viscose of the following composition was made:—

Cellulose	7.3 *per cent*
Sodium hydroxide	5.5 *per cent*
Carbon disulphide	33.0 *per cent* (on wt. of cellulose)
Sodium carbonate	4.1 *per cent*
Polyethylene glycol	1.2 *per cent*

The viscose was filtered and then 25 *per cent* acrylonitrile (based on the weight of cellulose) was stirred in, the viscose then being left to age for 13 hours. After ageing, it was spun at a ball fall viscosity of 60 seconds and a salt figure (sodium chloride test) of 12 into an aqueous coagulation bath comprising:—

Sulphuric acid	11.0 *per cent*
Zinc sulphate	2.0 *per cent*
Sodium sulphate	23.0 *per cent*

and at a temperature of 35°C. to form filaments of 6.2 d.tex/filament. The filaments were stretched in air by 25 *per cent*.

The filaments were then cut into staple fibres which were subjected to the usual neutralisation and washing procedures. On examination, the fibres were seen to have a substantially tubular structure, and the measured water imbibition was 211 *per cent*. This compared with a water imbibition of 125 *per cent* for fibres made identically by omitting the addition of acrylontrile, and a water imbibition of 90 *per cent* for solid fibres made using standard viscose process conditions.

Example 2

A viscose of the following composition was made:—

Cellulose	7.4 *per cent*
Sodium hydroxide	4.8 *per cent*
Carbon disulphide	33.0 *per cent* (on wt. of cellulose)
Sodium carbonate	3.0 *per cent*
Salt figure (sodium chloride test)	4.0
Ball fall viscosity	40.0 seconds

The viscose was fed to a spinning jet, and a 10 *per cent* aqueous solution of sodium polyacrylate was injected into the spinning line in weight proportion 1:26 to the viscose.

The injected viscose was spun into an aqueous coagulation bath comprising:—

Sulphuric acid	10.2 *per cent*
Zinc sulphate	0.8 *per cent*
Sodium sulphate	23.5 *per cent*

and at a temperature of 60°C. to form filaments of 1.7 d.tex/filament. The filaments were stretched in air by 25 *per cent*, cut into staple fibres and then given the usual neutralisation and washing treatments.

The cross-sections of the fibres were examined and were seen to be multi-limbed in various configurations and with a lumen in collapsed or partially collapsed form. The sodium polyacrylate content of the fibres was 4.9 *per cent* based on the weight of the cellulose, and their water imbibition was measured at 270 *per cent*. This compared with a water imbibition of 200 *per*

cent for fibres produced identically but without the sodium polyacrylate injection.

Example 3

The procedure of Example 2 was repeated but using poly (vinyl pyrrolidone) in concentration 4.9 *per cent* based on the weight of the cellulose instead of the sodium polyacrylate. The fibres produced were of similar cross-section and had a water imbibition measured at 280 *per cent*

Example 4

The procedure of Example 2 was repeated but using sodium carboxymethyl cellulose in concentration 2.0 *per cent* based on the weight of the cellulose instead of the sodium polyacrylate. The fibres produced were of similar cross-section and had a water imbibition measured at 294 *per cent.*

CLAIMS

1. Regenerated cellulose fibres comprising a cellulose wall defining a lumen, the cellulose wall incorporating substituent groups on the cellulose and/or a dispersed polymeric material, which substituent groups or polymeric material increase the liquid absorbency of the fibres.

2. Fibres as claimed in claim 1 having a water imbibition of at least 150 *per cent.*

3. Fibres as claimed in claim 1 having a water imbibition in the range 150 to 300 *per cent.*

4. Fibres as claimed in any of claims 1 to 3 which contain a dispersed polymeric material incorporated in the cellulose wall in an amount of less than 5 *per cent* by weight based on the weight of the cellulose.

5. Fibres as claimed in any of claims 1 to 4 which are substantially tubular in form with the lumen in expanded form.

6. Fibres as claimed in any of claims 1 to 4 in which the cellulose wall is of multi-limbed cross-section and the lumen is at least partially collapsed.

7. Fibres as claimed in any of claims 1 to 6 in which the cellulose wall incorporates a cellulose ether as dispersed polymeric material.

8. Fibres as claimed in claim 7 in which the cellulose ether is a carboxyalkyl cellulose.

9. Fibres as claimed in claim 8 in which the carboxyalkyl cellulose is sodium carboxymethyl cellulose.

10. Fibres as claimed in any of claims 1 to 6 in which the cellulose wall incorporates an alkali metal or ammonium salt of polyacrylic acid or an alkali metal or ammonium salt of a copolymer of acrylic acid and methacrylic acid as dispersed polymeric material.

11. Fibres as claimed in claim 10 in which the dispersed polymeric material is sodium polyacrylate.

12. Fibres as claimed in any of claims 1 to 6 in which the cellulose wall incorporates an N-vinyl amide polymer as dispersed polymeric material.

13. Fibres as claimed in claim 12 in which the dispersed polymeric material is poly(vinyl pyrrolidone).

14. Fibres as claimed in any of claims 1 to 6 in which the cellulose wall incorporates carboxyalkylated starch as dispersed polymeric material.

15. Fibres as claimed in any of claims 1 to 6 in which the cellulose wall incorporates poly(ethylene glycol) as dispersed polymeric material.

16. Fibres as claimed in any of claims 1 to 15 in which the cellulose has substituent carboxyethyl groups.

17. Fibres as claimed in any of claims 1 to 15 in which the cellulose has substituent hydroxyethyl groups.

18. Regenerated cellulose fibres as claimed in claim 1 and substantially as hereinbefore described in any of Examples 1 to 4.

19. An absorbent fabric or pad comprising regenerated cellulose fibres as claimed in any of claims 1 to 18.

20. An absorbent swab or dressing comprising regenerated cellulose fibres as claimed in any of claims 1 to 18.

21. A catamenial tampon comprising regenerated cellulose fibres as claimed in any of claims 1 to 18.

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